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Possible utilization of PET waste as adsorbent for Orange G dye removal from aqueous media

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ABSTRACT

In this study, easy and rapid adsorption of Orange G dye from aqueous solution using PET (polyethylene terephthalate) waste as low-cost adsorbent was examined. The optimal values of most important experimental parameters (initial solution pH, adsorbent dose and contact time) were established, in batch experiments, at room temperature (20°C ± 1°C). The kinetics of adsorption process follows the pseudo-second order kinetic model, and the Langmuir isotherm model provides the best fitting for the adsorption of Orange G dye onto PET waste. The maximum adsorption capacity, calculated in this case, was 6.9961 mg/g. Unfortunately, this value is quite small, and therefore an activation treatment of PET waste is necessary. The activation of PET waste with NaOH solution (4 N, 48 h) determines the increase of maximum adsorption capacity with 33.48%, indicating the formation of new active sites on adsorbent surface, probably due to the hydrolysis of some ester groups from superficial polymer chains. The adsorption capacities obtained for the Orange G adsorption onto activated PET waste from five different water samples does not differ significantly, which highlight the potential applicability of this adsorbent in the treatment of industrial effluents.

Keywords: Orange G dye; PET waste; Chemical activation; Isotherm and kinetic modelling

1. Introduction

The industrial effluents containing dyes discharged from various industries, such as textile dyeing and pigmentation, tanner, food, drug, pulp and paper, etc. [1,2], have negative impact on environment, because dyes drastically affects the quality of ecosystems [3]. This because, most of dyes, in addition to their coloration characteristics (which is noticeable even at very low concentration) [4,5], could cause the contamination of aquatic ecosystems and limited oxygenation of water surfaces. Therefore, even if the dyes toxicity is relatively low, the wastewaters containing dyes cannot be released into the environment without an adequate treatment.

The most widely used dyes, in industrial applications, are synthetic dyes, mainly due to their low cost, ease of preparation and high chemical stability in time [6,7]. Such synthetic dyes have in their structure one or more aromatic rings, which makes their degradation to require long period of time and increases the risk of toxic products formation, during such processes [8,9]. It is also the case of Orange G dye, which has numerous applications in textile dyeing industry [10], and which due to its low biodegradability and relative high solubility in water, is hazardous for environment and leads

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to negative consequences, when untreated effluents are discharged into water resources [11].

The methods used for the treatment of industrial effluents containing dyes, such as adsorption, chemical coagulation, photocatalytic degradation, membrane filtration, etc., [12–15] are based on well-known physico-chemical and/or biological processes [16], and are presented in literature as useful tools for the removal of organic dyes from industrial wastewater. Among these, adsorption is one of the most studied method which can be used for the removal of organic dyes from wastewater, mainly because it has high efficiency and low cost of operation. However, these advantages are closely related to the type of adsorbent material used in the removal processes. Therefore, besides the agricultural waste [16], other low-cost materials, such as clay minerals, aluminium oxide, fly ash, etc., [17–19], have been proposed in literature, for the adsorptive removal of organic dyes from aqueous effluents.

PET (polyethylene terephthalate) is a common synthetic polymer, which is widely used for the packaging manufacturing, due to its unique properties (such as good transparency, good malleability, high stability, high chemical resistance, low permeability to air or other atmospheric gases, etc.), [20]. Unfortunately, the use of PET as packing material, has led the discharge of huge amounts of PET packing waste, into environment. Because, the degradation of such plastic materials require a long period of time (over 180 years) [21], the recycling of PET wastes is the most economical way to control this pollution. Under these conditions, the finding of new possibilities for the valorisation of recycled PET waste is an actual issue, and their use as adsorbent materials can open new perspectives.

In this study, the PET waste was used as low-cost adsorbent for the easy and rapid adsorption of Orange G dye from aqueous solution. The optimal values of experimental parameters (initial solution pH, adsorbent dose and contact time) have been established in batch experiments. Pseudo-first order and pseudo-second order kinetics models, and Langmuir and Freundlich isotherm models were used for the mathematical modelling of the obtained experimental data. The activation of PET waste with NaOH solution was also considered, in order to increase the adsorptive characteristics of this low-cost material. The results included in this study have shown that PET waste can be an alternative for the designing of a new class of adsorbent materials for removing dyes from aqueous media.

2. Experimental setup

2.1. Materials

PET waste used as adsorbent in this study was obtained from recycling of PET bottles and was purchased from GreenFiber International Company (Iaşi, Romania). Before utilization in adsorption studies, the PET waste was washed with 1 N HNO_3 solution, in order to remove organic impurities, and then several times with distilled water. After drying, in air (at 50°C, 6 h), the adsorbent material was cut into pieces of about 1.0–2.0 mm. The activation of PET waste was done be treating 5 g of material with 25 mL of 4 N NaOH solution, at room temperature, for 48 h. After NaOH treatment, the activated PET waste was washed with distilled water, until a neutral pH, dried in air (at 50°C, 6 h), and used as adsorbent.

Orange G dye was purchased from Merck (Germany) and its chemical structure is illustrated in Fig. 1. The stock solution of Orange G dyes was prepared by dissolving 0.6 g of solid dye in 1,000 mL of distilled water, in a volumetric flask. The Orange G stock solution was kept at room temperature (20°C) and dark, for 24 h, to be sure that the solution is homogeneous before it is used in adsorption studies. All the working solutions were obtained from stock solutions by dilution with distilled water.

The initial pH of working solutions was adjusted using 0.1 N HNO_3 solution. All chemical reagents were of analytical degree and were used without purifications.

2.2. Adsorption experiments

Batch adsorption experiments were performed at room temperature (20°C ± 1°C) and have targeted the influence of the most important experimental parameters (initial solution pH, adsorbent dose and contact time) on the adsorption efficiency of Orange G dye onto PET waste.

Thus, 25 mL of Orange G solution, with a given value of initial pH, was intermittently stirred with different amount of PET waste, for specified time intervals. The concentration range of Orange G solution was 2.48–37.12 mg/L, variation range of initial solution pH was between 3.0 and 6.5, while the variation interval of adsorbent dose was between 4.0 and 20.0 g/L. Except the kinetic studies, where the contact time variation ranged from 5 to 180 min, in all others experiments, the contact time was 24 h.

After each adsorption experiment, the residual concentration of Orange G in aqueous solution (c, mg/L) was determined spectrophotometrically (Digital Spectrophotometer S 104 D, 1 cm glass cell, against distilled water, $\lambda = 495$ nm), using a prepared calibration curve.

The adsorption capacity (q, mg/g) and percentage of dye removal (R, %) were calculated using the following equations:

$$q = \frac{(c_0 - c) \cdot V}{m} \tag{1}$$

$$R = \frac{c_0 - c}{c_0} \cdot 100 \tag{2}$$

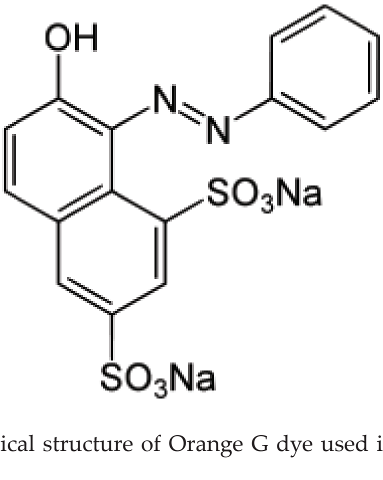


Fig. 1. Chemical structure of Orange G dye used in adsorption experiments.

where c_0 and c are the initial and residual concentration of Orange G dye in aqueous solution (mg/L), V is the volume of solution (L) and m is the mass of adsorbent (g).

The applicability of the activated PET waste for Orange G removal was tested using five different water samples, namely distilled water (obtained in laboratory, from a commercial distillation system), tap water (collected from University laboratories), drinking water (purchased from the supermarket), sea water (collected from Black Sea and preserved) and wastewater (obtained from a local company). All the water samples were filtered through quantitative filter paper and stored in dark. Each water sample was used to dilute 1.0 mL of Orange G solution (620 mg/L) to a volume of 25 mL, before to be mixed with 8 g/L activated PET waste, without to correct the initial pH. After 120 min of contact time, the concentration of Orange G dye was analyzed spectrophotometrically, as was described above.

FT-IR spectra of PET waste, before and after adsorption of Orange G dye, were recorded using a BioRad Spectrometer, in 400–4,000 cm^{-1} spectral domain, with a resolution of 4 cm^{-1} and 36 scans, by KBr pellet method. In the interpretation of FT-IR spectra, only absorption bands whose absorption maximums change by at least 5 cm^{-1} were considered.

2.3. Kinetics and isotherm models

The modelling of experimental data was performed using pseudo-first order and pseudo-second order kinetics models and Langmuir and Freundlich isotherm models, to highlight the most important aspects of the adsorption mechanism.

The mathematical equations of the pseudo-first order and pseudo-second order kinetic models, in their linear forms, can be written as follows [22,23]:

$$\log(q_e - q_t) = \log q_t - \frac{k_1}{2.303} \cdot t \tag{3}$$

$$\frac{t}{q_t} = \frac{1}{k_2 \cdot q_e^2} + \frac{t}{q_e} \tag{4}$$

where q_e and q_t are the adsorption capacities at equilibrium and at time t, respectively (mg/g), k_1 is the rate constant of pseudo-first order kinetics equation (min^{-1}) and k_2 is the pseudo-second order rate constant (g/mg min).

In case of adsorption isotherm models, the linear equations of Langmuir and Freundlich isotherm models can be expressed as follows [24,25]:

$$\log q = \log K_F + \frac{1}{n}\log c \tag{5}$$

$$\frac{1}{q} = \frac{1}{q_{\max}} + \frac{1}{q_{\max} \cdot K_L} \cdot \frac{1}{c} \tag{6}$$

where K_F is the Freundlich constant, n is a constant which characterizes the surface heterogeneity, $q_{\max}$ is the maximum adsorption capacity (mg/g), c is dye concentration at equilibrium (mg/L) and K_L is the Langmuir constant, related to the energy of adsorption process (L/g).

The best fit model for the description of the experimental data was selected on the basis of the values of regression coefficient (R^2), calculated using ANOVA statistical analysis.

3. Results and discussion

3.1. Optimization of adsorption conditions

Any adsorption process involves the realization of specific interactions between dye molecules from aqueous solution and active sites from adsorbent surface [26]. But, it is also well known that such interactions occur with maximum efficiency only in well-defined experimental conditions, which are directly responsible for the presence of a suitable speciation form of dye molecules, an adequate ionic state of superficial functional groups of adsorbent and an optimum ratio of the two (solid and liquid) phases [2,6,23]. Consequently, it is necessary to optimize the experimental conditions, in order to ensure the maximum efficiency of the adsorption process. The experimental parameters that should be considered for the optimization are: initial solution pH, adsorbent dose and contact time, because their values significantly influence the yields of the adsorption process. Fig. 2 illustrates the influence of each mentioned experimental parameter on the adsorption efficiency of Orange G dye onto PET waste.

Two observations were derived from the experimental results presented in Fig. 2(a). First, the adsorption capacity (q, mg/g) of PET waste increases when the initial solution pH increases from 3.09 to 5.39, after this pH range the q values remain constant. Second, on entire studied pH interval, the removal percentage of Orange G dye does not exceed 30%, and the increase of this parameter is insignificant (lower than 6%). The variation of the adsorption parameters (q and R) with the initial solution pH can be explained based on electrostatic interactions, relatively well described in the literature [27], considering that in the studied pH interval, the Orange G molecules are un-dissociated (pKa = 11.5) [26]. Thus, an initial solution pH of 6.5, which is the natural pH of Orange G aqueous solution, was considered optimum for this adsorption process, and was subsequently used. However, the experimental results from Fig. 2(a) highlighted two aspects that are important from practical point of view, namely (i) the process of removing Orange G by adsorption on PET waste, no rigorous control of the initial pH is required, which is a real advantage in the treatment of industrial effluents, and (ii) increasing the adsorptive performance of PET waste can only be done by increasing the number of superficial functional groups, and this can be achieved either by increasing the dose of the adsorbent or by activating it.

In order to check if the variation of adsorbent dose enhances the efficiency of Orange G adsorption onto PET waste, the influence of this parameter was also studied (Fig. 2(b)). It can be seen that increasing the adsorbent dose results in a decrease in adsorption capacity of PET waste from 11.31 to 2.73 mg/g. The decrease of q values is mainly determined by the decrease of ration between Orange G dye molecules and number of adsorption sites with increasing of adsorbent dose. Such variation is a typical one and, according with the studies from literature [28], indicates that only the superficial functional groups are involved in the adsorption process, whereas those inside of adsorbent particles remain inaccessible for interactions with the dye molecules. On the other hand, the values of removal percentages have a slightly increase (from 20.88% to 23.41%) on entire interval of adsorbent dose variation (Fig. 2(b)). This means that using a higher adsorbent dose is not a solution to improve the adsorption efficiency of Orange G dye onto PET waste adsorbent. Under these conditions, an adsorbent dose of 8.0 g/L was considered optimal for the adsorption of Orange G from aqueous solution onto PET waste.

Fig. 2(c) shows the influence of contact time on the adsorption of Orange G dye from aqueous solution onto PET waste. It can be observed that in the first 60 min, the adsorption process is relatively fast and more than 48% of initial Orange G molecules were retained in this time period. In the time interval between 60 and 120 min, the rate of adsorption process becomes slower, and reaches a plateau after 120 min of contact time. But, even if such variation of adsorption capacity as a function of contact time can be easily explained considering the number and availability of superficial sites from PET waste (which are higher at the beginning and decrease as the adsorption process occurs), the most important is that the contact time required to reach the equilibrium is 120 min. This proves that the adsorption of Orange G onto PET waste is a relatively fast process, which is an advantage for practical applications.

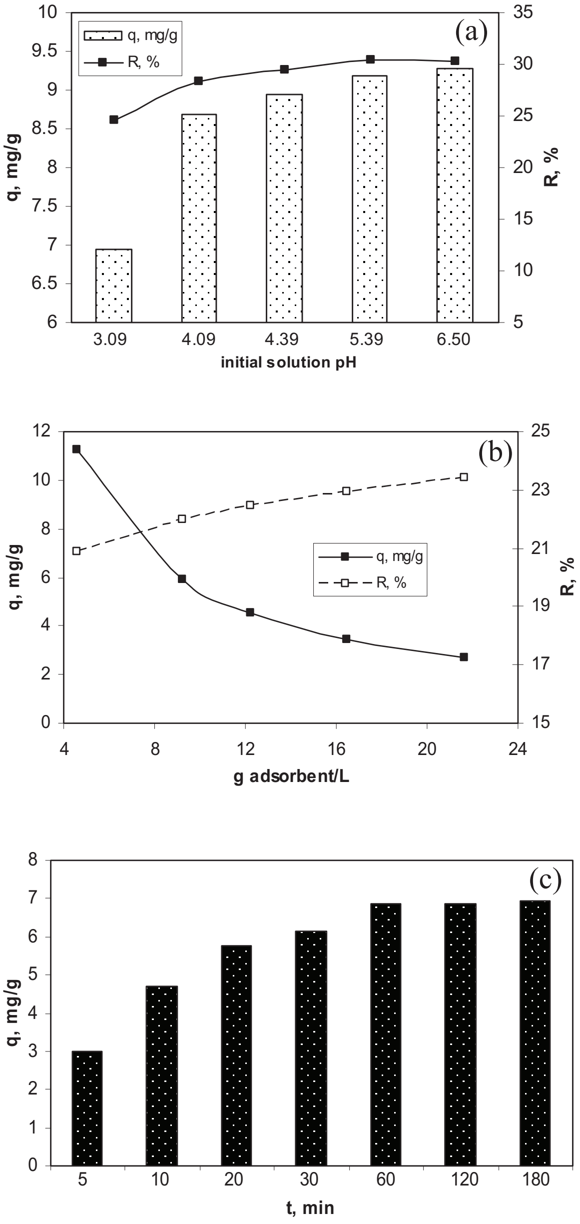


Fig. 2. Effect of experimental parameters on the adsorption efficiency of Orange G onto PET waste: (a) initial solution pH, (b) adsorbent dose and (c) contact time (experimental conditions: c_0 = 24.81 mg/L; temperature = 20°C).

Unfortunately in the optimal conditions, established experimentally step by step (initial solution pH of 6.5, 8.0 g adsorbent/L and 120 min of contact time), for a relatively low initial concentration of Orange G (24.81 mg/L), the removal percentage is not more than 50%. Therefore, the elucidation of the way in which Orange G molecules interact with PET waste will allow finding a suitable solution to increase the efficiency of the adsorption process. This can be done by kinetic modelling of experimental data.

3.2. Kinetic modelling of adsorption data

In the kinetic modelling of experimental data, two kinetic models, namely pseudo-first order model and pseudo-second order model, were used. In Fig. 3 are illustrated the linear

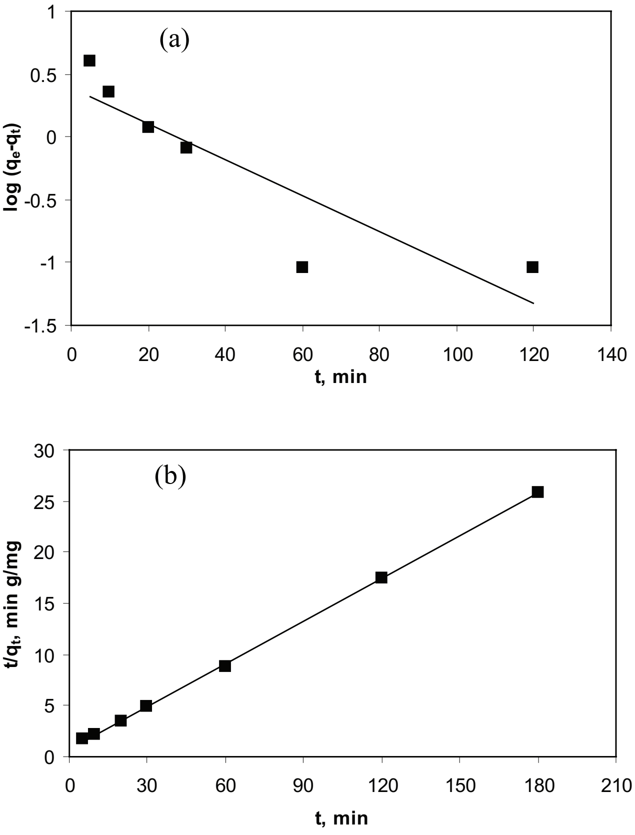


Fig. 3. Linear representation of the pseudo-first order kinetic model (a) and pseudo-second order kinetic model (b) for the Orange G adsorption onto PET waste, in optimal conditions.

representations of these two kinetic models for Orange G dye adsorption onto PET waste, and the most appropriate model was chosen based on regression coefficients (R^2), calculated from ANOVA statistical analysis. The kinetics parameters calculated for each kinetic model are summarized in Table 1.

It can be observed from Table 1 that the regression coefficient (R^2) is higher for the pseudo-second order kinetic model (0.9996), compared with the pseudo-first kinetic model (0.7942). This means that the pseudo-second order kinetic model is more appropriate for describing Orange G adsorption from aqueous solution onto PET waste, than the pseudo-first order model. Also, the applicability of pseudo-second order kinetic model in the description of experimental data is supported by the close values of q_e, calculated from the equation of pseudo-second order kinetic model (4.7161 mg/g) and obtained experimental (4.6954 mg/g).

Under these conditions, it can be considered that the Orange G adsorption onto PET waste is carried out through chemical interactions, which may be ion-exchange type or superficial complexation. In order to highlight the type of chemical interactions involved in adsorption process, FT-IR spectra of PET waste, before and after adsorption of Orange G dye, were recorded, as illustrated in Fig. 4.

The overlapping of the two spectra (Fig. 4) shows that the adsorption of Orange G dye does not cause major changes in the shape of FT-IR spectra of PET waste. Most of the absorption bands remain at the same maximum wave numbers, with two exceptions. Thus, the adsorption bands from 2,800 to 3,000 cm^{-1} and 1,400 to 1,050 cm^{-1} spectral regions, which correspond to C–H bonds from saturated alkyl radicals, and C=O from carbonyl and esters groups, are shifted after the adsorption of the dye. This indicates that the adsorption of Orange G molecules onto PET waste probably involves electrostatic interactions between ester groups of PET waste and –SO_3Na groups of dye (because in this way the steric hindrance is minimal), while the saturated alkyl radicals stabilize the Orange G molecules on PET waste surface through H-bonds with the other functional groups of adsorbent. Under these conditions, the efficiency of the adsorption process can only be increased by increasing the number of functional groups from PET waste surface, and this can be done by appropriate chemical treatment.

Table 1
Kinetic parameters obtained for Orange G adsorption onto PET waste

Pseudo-first order kinetic model		Pseudo-second order kinetic model	
R^2	0.7942	R^2	0.9996
q_e (mg/g)	0.4762	q_e (mg/g)	4.1761
k_1 (1/min)	0.0144	k_2 (g/mg min)	0.2702

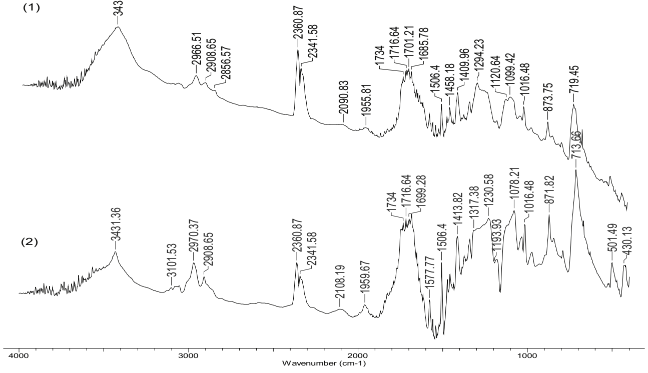
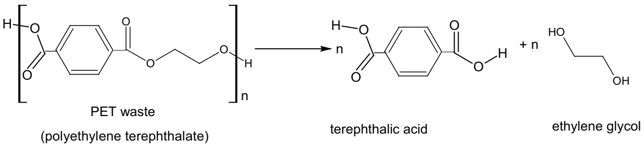


Fig. 4. FT-IR spectra of PET waste before (1) and after (2) adsorption of Orange G dye.

3.3. Activation of PET waste adsorbent

The chemical activation of PET waste was done with 4 N NaOH solution, at room temperature (20°C) for 48 h, and aimed to increase the number of adsorption sites on the adsorbent surface. It is expected that this alkaline reagent to hydrolyze the ester groups at least from superficial PET chains, making thus possible the apparition of new functional groups (mainly hydroxyl and carbonyl), according with the hydrolysis reaction:



The efficiency of activation treatment on the adsorptive performances of PET waste in the removal of Orange G from aqueous solution was examined in the initial dye concentration range between 2.48 and 37.12 mg/L, under optimal experimental conditions (initial solution pH of 6.5, 8.0 g adsorbent/L, 180 min of contact time, temperature of 20°C), compared with un-activated adsorbent (Fig. 5).

The experimental results illustrated in Fig. 5 show that after activation, the adsorption capacity of PET waste is increased, and this increase is more pronounced at high initial concentrations of Orange G dye. For the highest initial Orange G concentration (37.12 mg/L), the adsorption

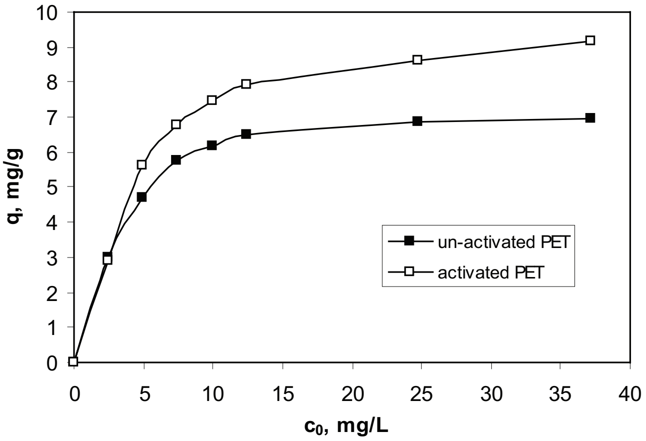


Fig. 5. Influence of activation treatment on the adsorptive performances of PET waste for Orange G dye from aqueous solution.

capacity of PET waste increases from 6.72 mg/g before activation to 9.21 mg/g after activation, which correspond to an improvement of adsorptive performances of this material by 33.48%. The obtained experimental results clearly show that after NaOH treatment the number of functional groups increases, and the activated material can retain more Orange G dye, from aqueous solution.

On the other hand, it should be noticed that if the un-activated PET waste reaches the saturation state at initial concentrations of Orange G higher than 12 mg/L (Fig. 5), after activation the saturation is not observed, even at initial dye concentration of 37 mg/L (Fig. 5). This is another argument that supports the hypothesis that, by activating PET waste with NaOH solution, the number of functional groups increases, which makes that the adsorptive performances of activated PET waste to be improved.

3.4. Isotherm modelling of equilibrium data

In order to have a quantitative measure of improving the adsorption capacity of PET waste, the experimental isotherms obtained before and after activation of adsorbent were modelled using Freundlich and Langmuir models. In each case the best fit isotherm model was chosen on the basis of the values of correlation coefficients (R^2), calculated with ANOVA statistical analysis. In Fig. 6 is presented a comparison between these two isotherm models and experimental data, in case of Orange G adsorption on un-activated and activated PET waste. Also, the isotherm parameters, calculated from linear representation of each isotherm model are summarized in Table 2.

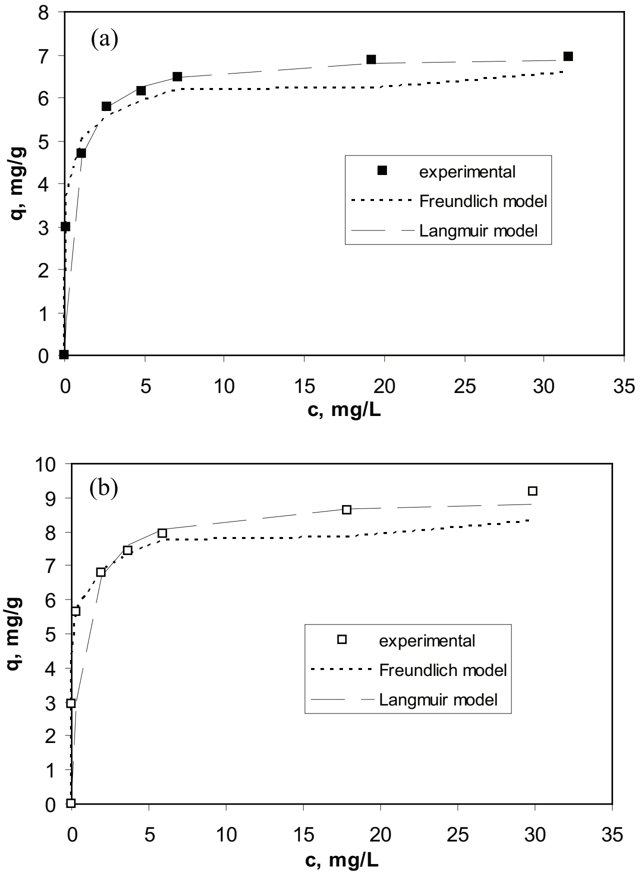


Fig. 6. Freundlich and Langmuir isotherms representation for Orange G adsorption onto PET waste (a) and activated PET waste (b).

Analyzing these data, it can be concluded that the Langmuir model best describes the experimental results obtained in case of Orange G adsorption onto PET waste, whether if the adsorbent is activated or not. This means that the adsorption of Orange G dye takes place at the exterior surface of adsorbent, until its particles are completely covered with dye molecules. As expected, the amount of Orange G required for the complete coverage is higher in case of activated PET waste ($q_{\max}$ = 8.9894 mg/g) than in case of un-activated PET waste ($q_{\max}$ = 6.9961 mg/g), and can be correlated with the number and availability of adsorption sites. Therefore, the increase in the number of superficial functional groups, mainly due to the activation, results in an increase in the number of dye molecules that can be retained on the surface of the adsorbent. These observations are supported and by the Freundlich isotherm parameters (n and K_F), which can be correlated with the intensity of adsorption process [25]. Higher values obtained of these parameters for activated PET waste (Table 2), clearly show that the intensity of adsorption process of Orange G dye has increased compared with un-activated PET waste. Whether is utilized activated PET waste or un-activated PET waste as adsorbent, the types of interaction between functional groups and Orange G dye do not change, and this is evidenced by the close values of Langmuir constants (K_L), which are correlated with the energy of adsorption process (Table 2).

However it should be noted that although this activation treatment improves the adsorptive performances of PET waste, the quantitative removal of Orange G dye from aqueous solution is not yet achieved.

In order to assess the efficiency of PET waste in the Orange G removal processes, the maximum adsorption capacity of this adsorbent, before and after activation treatment, was compared with the values obtained for other adsorbents, reported in literature (Table 3).

It can be observed that the values of $q_{\max}$ (mg/g) obtained for PET waste and activated PET waste in the removal of Orange G from aqueous media are comparable with those obtained for the adsorbents derived from inorganic materials, but are much lower than those obtained for different types of activated carbon. This suggests that, in order to improve the adsorptive performances of PET waste in dyes removal processes, the new treatments should be developed to increase the number of superficial functional

Table 2
Isotherm parameters of Orange G adsorption onto PET waste, before and after activation

Parameter	Langmuir model		Parameter	Freundlich model	
	Before	After		Before	After
R^2	0.9955	0.9853	R^2	0.8821	0.8933
$q_{\max}$ (mg/g)	6.9961	8.9894	$1/n$	0.1112	0.1082
K_L (L/g)	1.7948	1.5164	K_F (L/g)	0.4968	0.6382

Table 3
Comparative values of maximum adsorption capacities for Orange G removal on various adsorbent materials

Adsorbent	pH	$q_{\max}$ (mg/g)	Reference
Pumice	7.0	15.56	[11]
Nanoclay	4.0	39.40	[17]
Bagasse fly ash	4.0	18.79	[26]
Bentonite material	7.0	4.11	[29]
Moroccan phosphate rock	7.1	4.13	[30]
Thespesia populnea pods	–	9.13	[31]
Zinc oxide - activated carbon	2–10	153.85	[32]
PET waste	6.5	6.99	This study
Activated PET waste	6.5	8.99	This study

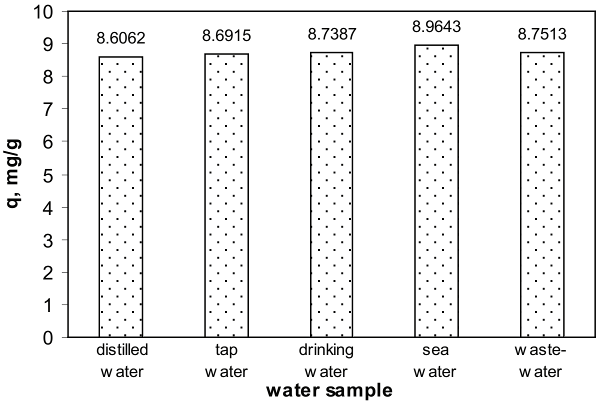


Fig. 7. Adsorption of Orange G dye from various water samples on activated PET waste.

groups per surface unit, and this will be in our attention in the future.

3.5. Water treatment applications

The potential applicability of activated PET waste as adsorbent for the removal of Orange G from aqueous media was tested using five types of water, collected from different sources. In each water sample, the concentration of Orange G was adjusted to 24.81 mg/L and was added 8 g/L of activated PET waste. The contact time was fixed at 120 min. Because the initial pH of water samples ranged between 5.12 and 6.30, there was no need to adjust the values of this parameter. The values of adsorption capacities were calculated using Eq. (1), and the obtained values are illustrated in Fig. 7.

It can be observed that compared with distilled water (8.6062 mg/g), the values of adsorption capacity obtained for the other water samples do not differ significantly. This indicates that the constituents of water samples do not interfere with the adsorption of Orange G adsorption. However, somewhat higher values of adsorption capacity obtained in case of sea water (8.9643 mg/g) and wastewater (8.7513 mg/g), where the salts content is higher than the tap water and distilled water is probably due to the decrease of the dissociation degree of $–SO_3Na$ groups from the Orange G molecules.

4. Conclusions

In this study, the PET waste was used as low-cost adsorbent for the easy and rapid adsorption of Orange G dye from aqueous solution. The batch experiments have examined the influence of most important experimental parameters (initial solution pH, adsorbent dose and contact time) on the Orange G dye adsorption efficiency. The optimal conditions, established step by step, were initial solution pH of 6.5, 8.0 g adsorbent/L and at least 120 min of contact time. Unfortunately, under these conditions at relatively low initial concentration of Orange G (24.81 mg/L), the removal percent is not more than 50%. The rate of adsorption process followed the pseudo-second order kinetic model, which highlights the importance of superficial functional groups in the adsorption process. Therefore an activation treatment of PET waste is necessary, in order to increase its adsorptive performances. This activation was done using 4 N NaOH solution, and the obtained adsorption data showed an increase of maximum adsorption capacity, calculated from Langmuir isotherm model, by 33.48%. Even if this activation treatment improves the adsorptive performances of PET waste, the removal percentage of Orange G dye remains relatively low, and in consequences further studies are still required. However, the adsorption capacities obtained for the Orange G dye adsorption onto activated PET waste from five different water samples does not differ significantly, which highlights the potential applicability of this adsorbent in the treatment of industrial effluents.

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